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C04B****(54) Process for water-repellent treatment of gypsum building units**

**(57) Water-repellent treatment of gypsum building units is achieved by processing hard wax or hard-wax-containing combinations with solid hydrocarbons with acid numbers >10 together with a metal salt characterised by better dissociation in respect of calcium sulphate and/or hydroxide and a sulphite lye or a modified sulphite lye with gypsum binder and water or di-hydrate and water within a mixing device so as to produce a mixed paste, whereupon the mixed paste is moulded and, if gypsum binder is used, dried, after hardening, at temperatures above the melting point of the wax used approximately to mass constancy, whereas if dihydrate is used the moulded strand of material is dehydrated in a saturated steam atmosphere at >130°C so as to form hemihydrate and is then hardened, in which case thermal treatment after hardening is unnecessary.**

**GB 2 205 562**

Process for water-repellent treatment of gypsum building units

5 The invention relates to a process for water-repellent treatment of gypsum building units by means of wax and wax-like products manufactured in preproduction plants and subjected during production to a thermal process.

10 With a view to water-repellent treatment of gypsum building units during the production process it has already been proposed in DD - WP 215 304 to make use of calcium sulphate hemihydrate with additions of hard wax or combinations with solid hydrocarbons containing hard wax, i.e. waxes with uncombined acid residues, mixed with water in solid,  
15 finely dispersed form and subjected after hardening of this mixture to thermal treatment. As distinct from previous water-repellent treatments - known and in part patented in this connection have been many different substances and substance mixtures such as silicones, paraffine-wax emulsion, ceresins, bitumen, resin emulsions, water-soluble silicates  
20 and stearates - this process has the advantage that owing to the presence of uncombined acid residues in these waxes compounds are formed in conjunction with the gypsum, which cannot dissolve in water, such compounds giving rise to a good long-time water-repellent effect after hardening and thermal treatment. A further advantage consists in the  
25 fact that the use of these waxes is physiologically safe, that in the mixture of water and gypsum they are virtually neutral and can, as solid granulates, be dosed in a simple manner.

However, this process is also characterised by the fact that a very high degree of water-repellency enabling use of the building unit in  
30 positively moist and wet rooms can be achieved only if relatively high amounts of water-repellent agents capable of melting down are added. In practice it is usual to add to the gypsum paste 4% wax in relation to the mass of gypsum binder. Since the waxes and products containing wax suitable for water-repellent treatment are very expensive, the  
35 material cost ratio when producing gypsum units treated in a water-repellent manner is too high, thus restricting, for economical reasons, application of this process only to mass-extensive gypsum building units, such as plasterboards.

Object of the invention

water-repellent treatment of gypsum building units by means of the above mentioned waxes such as hard wax, carnauba wax or combination with solid hydrocarbons containing hard wax, which are available in adequate amount, said process being superior to the known process by a more favourable ratio of the quantity of water-repellent agent and the degree of water-repellency, thus making it possible to produce water-repellent gypsum units more economically and thus to enlarge the scope of use and applicability of such units.

10 Description of the nature of the invention

The invention is intended to yield a process for the water-repellent treatment of gypsum building units on the basis of hard wax and hard-wax-like combinations with solid hydrocarbons, which makes it possible to increase the efficiency of said water-repellent agents and thus to produce water-repellent gypsum units using small amounts of wax in a manner advantageous as regards materials and costs.

According to the invention the object has been achieved by preparing a mixed paste by combining 1 to 4% hard wax or hard wax containing compounds with solid hydrocarbons with acid numbers  $> 10$  with a metal salt capable of better dissociation in respect of calcium sulphate and/or hydroxide such as potassium sulphate, aluminium sulphate, iron sulphate, sodium citrate, calcium hydroxide, ammonium sulphate and a sulphite lye or a technically modified sulphite lye with gypsum binder (hemihydrate) and water or with dihydrate and water, whereupon it is moulded. In the first case, if use is made of gypsum binder, hardening is followed by artificial drying roughly to the point of mass constancy, at temperatures higher than the melting point of the wax in use, as a rule  $> 60^{\circ}\text{C}$ . If dihydrate is used in the form of ground gypsum blocks or as an industrial by-product, produced e.g. by flue-gas desulphurisation, the moulded material is, prior to the hardening phase, dehydrated to hemihydrate in an autoclave containing saturated steam at temperatures  $> 130^{\circ}\text{C}$  so that at this technological stage the water-repellent agent system wax and additive already becomes fully active, as a result of which the strand of material is after dehydration already water-repellent and does not require any temperature treatment to this end after the following hardening stage. The highly water-repellent action of the combination of water-repellent materials used is due to the fact that the substances complementing the wax quickly dissociate so that, owing to the high availability of free ions optimal conditions exist for the

the water, which causes highly water-repellent saponification products to form, which settle within the gypsum matrix. In order to achieve this result it is sufficient to add 0.01% to 1% metal salt and/or hydroxide and 0.1 to 0.5% sulphite lye or technically modified sulphite lye. In this connection all percentages listed relate to the mass of the gypsum binder or dihydrate, whereas in the case of sulphite lye the concentration of solids constitutes the basis. In order to achieve maximum efficiency, metal salt, hydroxide and sulphite lye must be added dose by dose to the mixing water so as to cause predissolution.

The process according to the invention enables the production of water-repellent gypsum building units with a high degree of water-repellency. As distinct from the state of the art, the solution according to the invention makes it possible to save up to 50% wax while imparting to the gypsum units the same degree of water-repellency, thus reducing the total share of additive costs by about 25%. If the same amount of wax is used as heretofore, a considerably improved degree of water-repellency will be achieved, which is shown by the fact that building units according to the invention, which have been stored in water, absorb within a comparable unit of time and depending on the formulation only about 1/4 to 1/3 of the amount of water. This makes it possible to produce, subject to qualitative and application-technological points of view on the one hand and material-economic points of view on the other hand, gypsum building units with a variable degree of water-repellency and to extend their field of use.

#### Embodiments

The invention is explained in detail below with reference to embodiments.

#### Example 1:

ROMONTAN wax R 55, a wax mixture comprising 50% montan wax and 50% paraffine (hard wax-containing combination with solid hydrocarbons) with acid number 14 as well as of the substances listed in column 1 of the table are processed together with gypsum binder 05/82 and 70% water so as to produce a mixed paste, the metal salt and/or hydroxide as well as the sulphate lye being added, dose by dose, to the mixing water in the mixing vessel. The wax component is added in the form of granulate with a diameter of 0.8 mm together with the gypsum binder while the mixer is in operation. After hardening of the gypsum paste and drying at 70°C to roughly the point of mass constancy the water absorption after 30 minutes

indication of the degree of water-repellency. Column 3 contains values achieved when the wax component was used in accordance with the state of the art and without the other additives. The percentages of materials used as indicated in the table relate to the mass of gypsum binder. Lupoplast (thickened modified sulphite lye with about 20 to 25% solids) was used by way of sulphite lye modification.

10	Water-repellent gypsum building units produced by the process according to the invention (wax component + additives)	Water-repellent gypsum building units according to the state of the art (only wax component)	
	Percentages added to the gypsum paste	Water absorption after 30 minutes storage in water %	Water absorption after 30 minutes storage in water %
15	1	2	3
20	a) 2 % R55 1 % Lupoplast 1 % $K_2SO_4$	4.8	15.8
	b) 3 % R55 1 % Lupoplast 1 % $K_2SO_4$	2.7	10.1
	c) 4 % R55 1 % Lupoplast 1 % $K_2SO_4$	0.8	5.6
25	d) 2 % R55 1 % Lupoplast 0.01% sodium citrate	9.5	15.8
	e) 4 % R55 1 % Lupoplast 0.01% sodium citrate	3.4	5.6
30	f) 2 % R55 1 % Lupoplast 0.3% $Ca(OH)_2$	12.4	15.8
	g) 4 % R55 1 % Lupoplast 0.5% $Ca(OH)_2$	3.6	5.6

## Example 2

Calcium sulphate dihydrate is mixed with 50% water, 3% montan wax

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material, whereupon said strand of material is subjected to suctional dehydration until the content of residual water amounts to about 2%. This strand of material is autoclaved at about 140°C in an atmosphere roughly saturated with water vapour, which causes it to be converted into hemihydrate. This is followed in a normal atmosphere by re-hydratisation so as to form dihydrate. In the course of laboratory tests specimens were produced, which after 30 minutes storage in water exhibited a water absorption of < 1%.

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## Claims

1. Process for water-repellent treatment of gypsum building units, in the process of production of which gypsum binder with additives of hard wax or combination with solid hydrocarbons containing hard wax, i.e. waxes with uncombined acid residues, are processed in solid, finely dispersed form with water, whereupon they are moulded and the wax component is caused by thermal processes to melt down, characterised in that, related to the mass of gypsum binder or dihydrate, 1 to 4% hard wax or hard wax-containing combinations with solid hydrocarbons with acid numbers  $> 10$  are made together with a metal salt characterised by a better dissociation in respect of calcium sulphate and/or hydroxide such as potassium sulphate, aluminium sulphate, iron sulphate, sodium citrate, calcium hydroxide, ammonium sulphate and a sulphite lye or a technically modified sulphite lye with gypsum binder and water or with dihydrate and water within a mixing device so as to produce a mixed paste, whereupon the mixing paste is moulded and, in the first case where use is made of gypsum binder, dried after hardening at temperatures above the melting point of the wax, as a rule  $> 60^{\circ}\text{C}$ , to approximately the point of mass constancy, while in the second case, where use is made of dihydrate, the moulded strand of material is dehydrated in an atmosphere of saturated steam at  $> 130^{\circ}\text{C}$  so as to produce hemihydrate, whereby in this stage the water-repellent agent system wax and additives already becomes fully active and no thermal treatment to this end is required any more after subsequent hardening.
2. Process for water-repellent treatment according to claim 1, characterised in that, in relation to the mass of gypsum binder or dihydrate, 0.01% to 1% metal salts and/or hydroxide are used.
3. Process for water-repellent treatment according to claims 1 and 2, characterised in that use is made of 0.1 to 0.5% sulphite lye or technically modified sulphite lye, related to its ratio of solids in respect of the gypsum binder or dihydrate.
4. Process for water-repellent treatment according to claims 1 to 3, characterised in that the metal salt, the hydroxide and the sulphite lye are added, dose by dose, to the mixing water and predissolved.



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5. A process for water-repellent treatment of gypsum containing building units, wherein the units are made from a paste including gypsum or dyhydrate binder, hard wax or hard wax containing combinations with solid hydrocarbons with acid numbers  $> 10$  together with a metal salt or hydroxide, a sulphite lye and water, and after hardening are subjected to thermal process which causes melting of the wax.
6. A process for water-repellent treatment of gypsum containing building units, substantially as herein described with reference to the Example.